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## Evaluation of Thermophysical Properties of TiN Nanofluid Using Combined Experimental-Statistical Approach

Kishor Deshmukh<sup>1,2</sup>, Dr. Suhas Karmare<sup>3</sup>

<sup>1</sup>Research Scholar, KJ's Educational Institute, Trinity College of Engineering and Research, Pune

<sup>2</sup>Assistant Professor, Amrutvahini College of Engineering, Sangamner

<sup>3</sup>Government College of Engineering, Avasari

dkishor21@gmail.com

### **ABSTRACT**

Due to their enormous thermophysical properties, nanofluids open a new dimension in solar thermal applications. The preparation of stable, efficient, and low-cost nanofluids is an emerging area of research. According to NIMS research, titanium nitride (TiN) nanoparticles have LSPR properties. It enables a superior photoabsorption feature. To prepare distilled water-based nanofluid at 0.025%, 0.05%, 0.05%, 0.075%, and 0.1% concentrations, distilled water-based nanofluid at 0.025%, 0.05%, and 0.1% concentrations were chosen. The thermal conductivity and viscosity of TiN nanofluids and base fluid are measured experimentally at temperatures ranging from 30°C to 55°C. Determination of thermal conductivity and viscosity of nanofluid through experimentation is cumbersome. The present study deals with thermal conductivity and viscosity modeling of water-based stable plasmonic TiN nanofluid using the surface response method. ANOVA determine the significance of input variables and their interactions. The performance of predictive models was measured in terms of correlation coefficient and mean square error to acknowledge the best fit. The best-fitted RSM model used to predict thermal conductivity and viscosity. The surface response method gives reliable and efficient model results for thermal conductivity and viscosity with an accuracy of 99.47% and 99.97%.

Keywords: Nanofluid, TiN; Thermal Conductivity; Viscosity; RSM; Prediction;

## 1. INTRODUCTION

According to the population division of the department of economic and social affairs, the world's population is increasing dramatically by leaps and bounds. The industrial sector is expanding day by day to fully meet energy needs. Fossil fuel is the most common energy source. The continuous depletion and scarcity of fossil fuels motivate researchers to find renewable energy sources. Solar energy is the only renewable energy source on the planet capable of meeting energy demands without polluting the environment [1][2][3]. Solar thermal collectors are used to harvest solar energy. Solar collectors absorb radiation and transfer the absorbed energy to working fluid [1][2]. Conventional non-dielectric fluids like water, ethylene glycol, oils, and dielectric fluids like aliphatic liquids, silicone liquids, and fluorocarbons suffer from poor thermophysical properties. These properties limit the thermal performance of solar thermal collectors [1]. Choi (1995) (Argonne National Laboratory, USA) developed a nanofluid by dispersing nanosize (100nm) particles in the base fluid to enhance base fluid properties [4]. Hybrid nanofluids are the next version of nanofluids, prepared by suspending two or more types of nanoparticles in the base fluid [1]. Nanomaterials exhibit distinct thermal, physical, chemical, and mechanical properties due to the presence of more atomic atoms present on grain borders and a higher surface area to volume ratio. The nanofluid with tunable thermophysical properties will be the next-generation fluid.

Nanofluid thermophysical properties depend upon the nature of the base fluid, particle type, temperature volume concentration, size, and shape of nanoparticles. Thermal conductivity and viscosity of nanofluid have a substantial effect on heat transfer performance and are hence considered the most vital properties [8]. In addition to heat transfer properties, the rheological behavior of nanofluids needs to be investigated simultaneously [9]. The thermal conductivity and viscosity of nanofluid are a function of temperature, volume concentration, and stability parameters. Accurate prediction of thermophysical properties is not possible by conventional models. See computing techniques like RSM, ANN, NSGA, CFD, Particle swarm Technology, and fuzzy logic reduce experimentation costs and time [7]. An optimum volume concentration is required to design a sustainable and did multi-objective optimization of thermal oil-based MWCNT nanofluid to maximize thermal conductivity and minimize viscosity. RSM tools were used to develop a predictive model.

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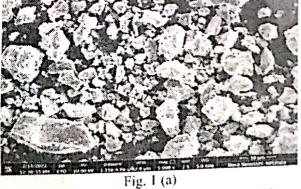
In addition to the prediction, a detailed optimization study was conducted for four different cases according to applications. Maqsood et al. [13] investigated the thermophysical properties of nanofluid (flyash + water) using the RSM tool. An empirical model was developed and tested with experimental data. The aim was to determine the optimum volume concentration and thermophysical characteristics. Amir et al. [15] optimized the geometrical parameters and flow characteristics of water-based Al2O3 nanofluid inside a corrugated heat exchanger using a genetic algorithm. Al2o3 nanofluid acts as a cooling fluid. He considered corrugation length, depth, phase shift angle, nanoparticle concentration, and Reynold's number for optimization. MATLAB is a computational language used for modeling. The fluid viscosity plays a crucial role in the micro convection phenomenon, hence considered a vital aspect while studying nanofluid.

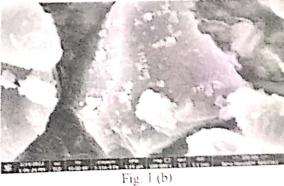
The thermal conductivity and viscosity of nanofluid increase with the volume concentration of nanoparticles[7]. The increase in thermal conductivity causes heat transfer enhancement. An increase in viscosity increases pressure drop and decreases heat transfer rate. Due to high experimentation costs and time consumption, researchers are trying to develop a new modeling technique have the ability to predict the thermal conductivity and viscosity of emerging nanofluids without physical experimentation. The effect of volume concentration (0–0.1%) and temperature (30–55°C) on the thermophysical properties of TiN nanofluid was studied. The scope found for prediction of TiN nanofluid thermal conductivity and viscosity using the RSM method [17].

# 2. NANOFLUID PREPARATION, CHARACTERIZATION AND EXPERIMENTAL INVESIGATION OF THERMOPHYSICAL PROPERTIS

The volume concentration and temperature are the most significant factors which affect the thermal conductivity and viscosity of nanofluid, thereby influencing system heat transfer performance [7]. The nanoparticles of less than 100 nm give better stability and heat transfer performance, for this research, average of 50 nm-sized nanoparticles used [18]. The nanoparticles start to agglomerate beyond a 0.10% volume concentration. So, five concentration levels selected (0%, 0.025%, 0.050%, 0.075%, and 0.1%) for our investigation. The temperature of the nanofluid inside the solar thermal collector varied from 30°C to 60°C from 9.00 am to 5:00 pm. Hence, five temperature levels (35°C, 40°C, 45°C, 50°C, 55°C) are considered. The thermal conductivity and viscosity of TiN nanofluid were measured experimentally using an L25 orthogonal array.

TiN nanoparticles consider for this research work because of LSPR unique feature. Commercially available TiN nanoparticles (40-50nm)[19] purchased from Intelligent materials Pvt. Ltd., Punjab (India). Its mass fraction purity exceeds 99.9%. The water was distilled and subsequently treated by a Milli-Q water purification system (Pure Lab Flex 3, ELGA UK) used as the base fluid for experimentation. The surfactants enhance the stability of nanofluids [7]. Surfactant converts the hydrophilic surface of the nanoparticles to hydrophobic and vice versa. If base fluid contains O-H bond (polar solvent), it's better to select water soluble surfactants (Sulphate, Sulphonate, Fatty Amines, Fatty Acid Alcohols) [20][21]. LOBA Chemie Pvt. Ltd. branded extra pure needle shape sodium lauryl sulfate (SLS) used as surfactant. Morphological structure of TiN particles analyzed by FESEM technique. Fig.1 (a) and 1 (b) are SEM images of TiN nanoparticles captured by FESEM (FEI Nova NanoSEM 450, SPPU, Pune). Nanoparticles high-resolution images captured at 10μ and 500nm scale at 100,000× magnification. These images show TiN nanoparticles have agglomerates, and it needs sonication. Average particle size matches with supplier's test reports.





ig. 1 (a)
Fig. 1 FESEM of TiN Nanoparticle at 10μ and 500 nm Scale

Nanofluid preparation is a crucial stage in nanofluids research. To achieve effectiveness in terms of heat transfer two-step method is followed. TiN nanoparticles were dispersed into distilled water (1000ml) with the volume fractions of 0.025%, 0.050%, 0.075%, 0.1%[19]. The law of the mixture formula gives the quantity of volume fractions of 1/10th of nanoparticles to improve its stability. Calculated amount of dry titanium nitride powder concentration of 1/10th of nanoparticles to improve its stability. Calculated amount of dry titanium nitride powder and sodium lauryl sulfate (SLS) was weighed using a precision balance (Shimadzu AUX220) and poured into the and sodium lauryl sulfate (SLS) was weighed using a precision between TiN nanoparticles agglomerates nanoparticles.

The magnetic stirrer (Remi 1MLH) breaks the powerful Vander wall attraction forces between nanoparticles. The Magnetic stirrer operates at 1000 rpm for 60 min. All the samples sonicate for 120 min in a bath type ultrasonic stirrer at room temperature. The Probe-type ultrasonicator was used at the last stage to obtain homogenous and stable nanofluid. Nanofluid characterization is measured in terms of stability and particle size. Stability indicates the nanoparticle rate of aggregation in the base fluid. The prepared TiN nanofluid is visually stable after 15 days. To measure the zeta potential and particle size of TiN nanofluid SZ-100 HORIBA instrument of Amrutvahini College of Pharmacy sangamner used. The zeta potential value is the function of time. Strong negative zeta potential indicates TiN nanoparticles do not tend to come together. The zeta potential values are 0, -61.6 mV,-61.1 mV, 37.1mV, and 30.1mV respectively. The particle size are 100.0 nm,117.3 nm, 141.5 nm, 144.3nm, and 151.1 nm respectively. Thermal conductivity and viscosity considered as important thermophysical property.

Thermal conductivity is the ability of the material to conduct heat. The uniform dispersion of nanoparticles in the base fluids influences the thermal conductivity of the base fluids. The Brownian movement of nanoparticles at the nanoscale level is considered a key mechanism for the micro convection in a nanofluid. Transient hot wire apparatus developed by Dr. Sonawane and Prof. Kokate at Sapat College of Engineering, Nashik. According to him, the setup gives accurate results by eliminating natural convection effects. Thermal conductivity of TiN nanofluid measured on his experimental apparatus. The transient-hot-wire method involves measuring the slope of the rise in the temperature of platinum wire against the logarithmic time interval to calculate the effective thermal conductivity of the nanofluids [7][22]. The deionized water, air, and toluene used to calibrate the instrument. After calibration of transient hot wire apparatus TiN nanofluid thermal conductivity of measured.

The viscosity is a property of the fluid which offers resistance to the movement of one layer to the adjacent layer. The viscosity of nanofluid increases with % volume concentration. Nanofluid viscosity variation depends upon volume concentration, range of shear stress rate selected, base fluid type, agglomeration rate, temperature, purity, size, shape, type of nanoparticles, preparation, and dispersion method. The viscosity of TiN-Water nanofluid was measured using Brookfield Viscometer DV-I Prime (SPPU, Chemistry department) at maximum % torque and 50rpm with accuracy ±0.1.

#### 3. RSM BASED PREDICTIVE MODEL

Surface response methodology is the most popular statistical and mathematical tool. It develops an empirical relationship between inputs and output variables considering the multiple regression technique. The RSM concentrates on the effect of the least significant input variable on the response variable[13]. The main aim of the RSM is to develop thermal conductivity and viscosity predictive model. The % volume concentration and temperature were considered input variables and thermal conductivity and viscosity as the response variable. A second-order predictive model was developed by considering the mutual relationship between input and response variables.

$$Y = \beta_0 + \sum_{i=1}^p \beta_i X_i + \sum_{i=1}^p \beta_{ii} X_i^2 + \sum_{i=1}^{p-1} \sum_{j=1}^p \beta_{ij} X_i X_j$$
Where  $\beta_0$ ,  $\beta_{1i}$ , and  $\beta_{1j}$  are coefficient of the regression model.  $X_i$ , and  $X_j$  are independent variables and  $Y$  the

Where  $\beta_0$ ,  $\beta_{11}$ , and  $\beta_{1j}$  are coefficient of the regression model.  $X_1$ , and  $X_j$  are independent variables and Y the denotes dependent response variable. At the last stage, ANOVA is used to investigate the importance of the predictive model and input variables. The ANOVA calculates the degree of freedom, the number of squared deviations, and the mean square error for each input. The input will consider as significant if the p-value is < 0.05.

$$K = 0.6132 + 1.817A + 0.00157B - 4.693A^2 - 0.000005B^2 + 0.00466AB$$
 (2)

Eq. (2) gives the predictive model for thermal conductivity considering the interaction between input variables. Whereas K is thermal conductivity, A and B denote % volume concentration temperature. The F value of the model is 732.58, and the p-value is 0.000. It shows the model is significance and acceptability of the developed model. The R2 value is 99.48%. The thermal conductivity model shows % volume concentration, temperature, and square of % volume concentration are significant factors for thermal conductivity enhancement. Interaction between % volume concentration and the temperature does not contribute to enhancement.

$$\mu = 1.410 + 3.41A - 0.0173B - 1.6A^2 - 0.000034B^2 + 0.0496AB$$
(3)

Eq. (3) gives the viscosity predictive model considering the interaction between input variables. Whereas  $\mu$  is viscosity, A and B denote % volume concentration and temperature. The F value of the model is 127.37, and the p-value is 0.000. It shows the significance and acceptability of the developed model. The R2 value is 97.11% The viscosity model shows % volume concentration and temperature are significant factors for viscosity enhancement. Individual and mutual interaction between % volume concentration and the temperature does not contribute to enhancement. The % contribution of each input variable, its square and mutual interaction between them measured from ANOVA. The most significant input variable is % volume concentration (A) followed by temperature (B) for thermal conductivity enhancement and temperature (B) followed by  $^{0}$  volume concentration (A) for viscosity enhancement. Fig. 2 and 3 show a scatter plot of experimental thermal conductivity and viscosity value with predicted values. The R2 value of both models is between 97-98 %. Higher R2 values show the reliability and efficiency of predicted models.

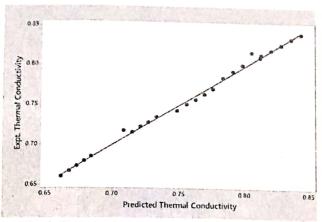


Fig. 2 Scatter plot of Experimental and RSM Predicted Thermal Conductivity of TiN Nanofluid.

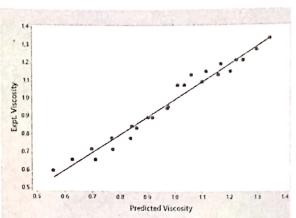


Fig. 3 Scatter plot of Experimental and RSM Predicted TiN Nanofluid Viscosity.

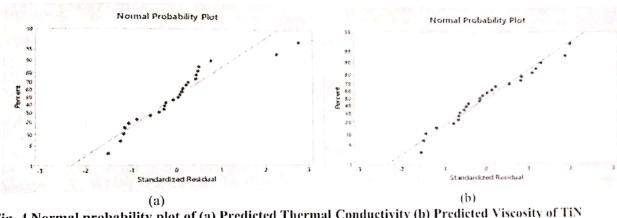


Fig. 4 Normal probability plot of (a) Predicted Thermal Conductivity (b) Predicted Viscosity of TiN After the model development adequacy test and outlier plot test were performed. Fig. 4 (a) & Fig. 4 (b) show residual plots for thermal conductivity and viscosity model. Results are near to the straightened line, randomly distributed, and not following any pattern. It confirmed it is suitable for optimization and prediction. The data points which do not follow the trend of other data points are called an outlier. Detection and removal of outliers is an important step while the development of the predictive model. Fig.5 shows an outlier plot for thermal conductivity and viscosity. All the data points fall inside the allowable range of the predictive model. Outlier plots confirm thermal conductivity and viscosity predictive models are reliable and give correct predictions.

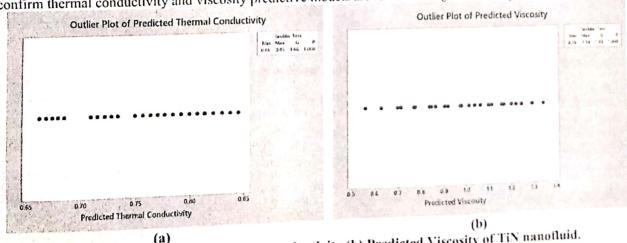
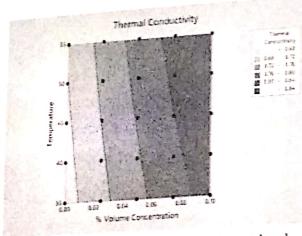
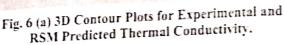


Fig. 5 Outlier plot of (a) Predicted Thermal conductivity (b) Predicted Viscosity of TiN nanofluid.

The contour surface response plot provides the influence of each parameter on thermal conductivity enhancement. The contour surface response plot provides the influence of each parameter on thermal conductivity enhancement. Fig. 6 (a) shows thermal conductivity of TiN nanofluid increases with an increase in % volume concentration and 55°c, temperature. Maximum thermal conductivity of 0.84 W/mk obtained at 0.1 % volume concentration and 55°c.





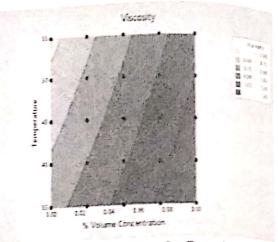


Fig. 6 (b) 3D Contour Plots for Experimental and RSM Predicted Viscosity.

Fig. 6 (b) shows Viscosity of TiN nanofluid increases with an increase in % volume concentration and decreases with temperature. Minimum viscosity of 0.6 cP obtained at 0 % volume concentration and 55%

# 4. COMPARATIVE ERROR ANALYSIS OF RSM MODEL AND EXPERIMENTAL RESULT

Comparative error analysis of RSM model and experimental results performed in terms of correlation coefficient (R2), root mean square (RMSE) standard error of prediction (SEP), and average absolute deviation (AAD). Maximum R2 shows the closeness of data points to the regression line. The minimum MSE signifies how data points are far from the regression line. The experimental and predicted values of thermal conductivity and viscosity compared. The result indicates strong agreement with minimum % error. The R2 value of the RSM thermal conductivity model is 99.48% and 96.97% respectively. It indicates RSM thermal conductivity model predicts closer accurate data. The R2 value of the RSM thermal conductivity model is 97.81%. To predict reliable and efficient results RSM thermal conductivity and viscosity model are suggested.

This paper gives an overview of the preparation, characterization, determination, and prediction of thermal conductivity and viscosity of stable TiN nanofluid. Prepared TiN nanofluid shows long-duration stability and enhanced thermophysical properties. The thermal conductivity and viscosity determinations are time-consuming and costly. The RSM statistical tools develop a predictive model with volume concentration and temperature as input parameters. A predictive model enables the selection of process parameters. The thermal conductivity and viscosity predictive models are functions of volume concentration and temperature using the surface response method and an artificial neural network.

method and an artificial nethod. 
$$K = 0.6132 + 1.817\varphi + 0.00157T - 4.693\varphi^2 - 0.000005T^2 + 0.00466\varphi T$$

$$\mu = 1.410 + 3.41\varphi - 0.0173T - 1.6\varphi^2 - 0.000034T^2 + 0.0496\varphi T$$

The RSM thermal conductivity and viscosity model gives reliable, accurate results with 99.47% and 99.97% accuracy. The research focused on the prediction of thermal conductivity and viscosity. There is scope for more than one thermophysical property prediction using different intelligent techniques. The optimum volume concentration and temperature are helpful for maximum thermal conductivity and minimum viscosity determination in the future. These optimum conditions will reduce cost and system size. TiN nanofluid worthings for solar applications discovered.

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